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Chemical Information From GC-MS Studies of Ethanol Extract of *Andrographis Paniculata* and Their Corrosion Inhibition Potentials on Mild Steel in HCl Solution

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GC-MS study of ethanol extract of *Andrographis paniculata* revealed the presence of hydrocarbons ((E)-tetradec-3-ene), alcohol (6-methyl heptan-1-ol), aromatics (undecan-5-ylbenzene, dodecan-5ylbenzene, undecan-2-ylbenzene, (2-methylundecan-6-yl) benzene and dioctyl phthalate). Some of the identified compounds were found to be green raw materials for polymer industries and pharmaceutical companies. Corrosion inhibition potentials of the ethanol extract of *Andrographis paniculata* was also investigated and it was found that the extract inhibited the corrosion of mild steel in HCl solution through the mechanism of physical adsorption. Quantum chemical approaches have been used to investigate the active components of the extract that are responsible for the inhibition of the corrosion of mild steel in HCl solution. The results obtained, indicated that the most active ingredient responsible for the corrosion inhibition process is pentadecanoic acid. Other compounds with lesser activity include 6-methyl hepta-1-ol, methyl palmitate, methyl icosanoate, methyl stearate, oleamide, dicotyl phthalate and ethyl docosanoate are the active.

Keywords: Corrosion, inhibition, *Andrographis paniculata*, GC-MS, quantum study

1. INTRODUCTION

In fertilizers, petroleum, metallurgical and other industries, corrosion of metallic components are likely during acid cleaning, pickling, etching and related processes [1-3]. Although there are several options in controlling corrosion of metals, the use of inhibitors has been proven to be one of the best options. Most inhibitors are organic compounds whose inhibition potentials can be correlated with

their chemical structures. However, some of them are toxic, non degradable and are not eco-friendly [4].

Extract of plants and other natural products have also been utilized for the protection of metals against corrosion [5-8]. Researches generally agreed that most of these plant extracts are green corrosion inhibitors because they are biodegradable, less toxic and do not contain heavy metals [9]. In the light of these, several plants extracts have been investigated and their corrosion inhibition properties are often attributed to its phytochemical constituents [10]..

The active phytochemicals in plants that are effective for corrosion inhibition can be regarded as those that have hetero atom(s) in their aromatic or long carbon chain. Possession of π-electrons or suitable functional groups may also facilitate the transfer of charge from the inhibitor's molecule to the charged metal surface (physical adsorption) or the transfer of electron from the inhibitor's molecule to vacant d-orbital of the metal (chemical adsorption). Therefore, in order to identify the active constituents of plant extract involved in corrosion inhibition, explicit knowledge of the chemical structures of its phytochemicals is required. Therefore, the present study is aimed at elucidating the chemical structures of ethanol extract of *Andrographis paniculata* and to investigate their corrosion inhibition potentials for mild steel in solutions of HCl (using gravimetric and gasometric methods). Also, from the identified chemical constituents or structures that are inherent in ethanol extract of *Andrographis paniculata*, other industrial potentials of the plant shall be investigated. FTIR shall be used to study the functional groups associated with the adsorption of the inhibitor. Finally, quantum chemical parameters shall be used to predict the order of importance of the various constituents of the extract in the corrosion inhibition process.

2. EXPERIMENTAL

2.1. Materials

Materials used for the study were mild steel sheet of composition (wt %) Mn (0.6), P (0.36), C(0.15) and Si (0.03) and Fe (98.86). The sheet was mechanically pressed cut into different coupons, each of dimension, 5 x 4 x 0.11 cm. Each coupon was degreased by washing with ethanol, cleaned with acetone and allowed to dry in the air before preservation in a desiccator. All reagents used for the study were Analar grade and double distilled water was used for their preparation. Concentrations of HCl prepared for gasometric, thermometric and weight loss studies were 2.5, 2.5 and 0.1 M respectively.

2.2. Extraction of plants

Samples of *Andrographis paniculata* leaves were obtained locally. The leaves were sun-dried, ground and soaked in a solution of ethanol for 48 hours. After 48 hours, the samples were cooled and filtered. The filtrates were subjected to evaporation at 352 K in order to leave the sample free of

ethanol. The stock solutions of the extract so obtained, were used in preparing different concentrations of the extract by dissolving 0.1- 0.5 g of the extract in 1 L solution of 0.1 M of HCl.

2.3. Gravimetric method

In the gravimetric experiment, a previously weighed mild steel coupon was completely immersed in 250 ml of the test solution in an open beaker. The beaker was covered with aluminium foil and maintained at 303 K. After every 24 hours, the corrosion product was removed by washing each coupon (withdrawn from the test solution) in distilled water, containing 50 % NaOH and 100 g l^{-1} of zinc dust. The washed coupon was rinsed in acetone and dried in the air before re-weighing. The experiment was repeated at 333 K. In each case, the difference in weight for a period of 168 hours was taken as the total weight loss. From the average weight loss (mean of three replicate analysis) results, the inhibition efficiency (%I) of the inhibitor, the degree of surface coverage (θ) and the corrosion rate of mild steel (CR) were calculated using equations 1, 2 and 3 respectively [11];

$$\%I = (1 - W_1/W_2) \times 100 \quad (1)$$

$$\theta = 1 - W_1/W_2 \quad (2)$$

$$CR = \Delta W/At \quad (3)$$

where W_1 and W_2 are the weight losses (g) for mild steel in the presence and absence of the inhibitor, θ is the degree of surface coverage of the inhibitor, A is the area of the mild steel coupon (in cm^2), t is the period of immersion (in hours) and ΔW ($\Delta W = W_2 - W_1$) is the weight loss of mild steel after time, t.

2.4. Chemical analysis

Phytochemical analysis of ethanol extract of *Andrographis paniculata* leaf was carried out according to the method reported by Odiongenyi *et al*. [12]. Frothing and Na_2CO_3 tests were used for the identification of saponin. Bromine water and ferric chloride tests were used for the identification of tannin. Leberman's and Salkowski's tests were used for the identification of cardiac glycodises while Dragendorf, Hagger and Meyer reagents were used for the identification of alkaloid.

2.5. Quantum chemical calculation

Full geometry optimization was achieved using MM2 and DFT programmes in the Hyperchem release 10 computational software. Single point energy calculations were carried out using PM6 Hamiltonian in the MOPAC 2008 software for Windows. Calculations were performed on an IBM compatible Intel Pentium IV (2.8 GHz, 4 GB RAM) computer. The following quantum chemical

indices were calculated: the energy of the highest occupied molecular orbital (E_{HOMO}), the energy of the lowest unoccupied molecular orbital (E_{LUMO}) and the energy gap ($\Delta E = E_{LUMO} - E_{HOMO}$).

3. RESULTS AND DISCUSSIONS

3.1. GC-MS study

Figure 1 shows the GC-MS spectrum of ethanol extract of Andrographis *paniculata*.

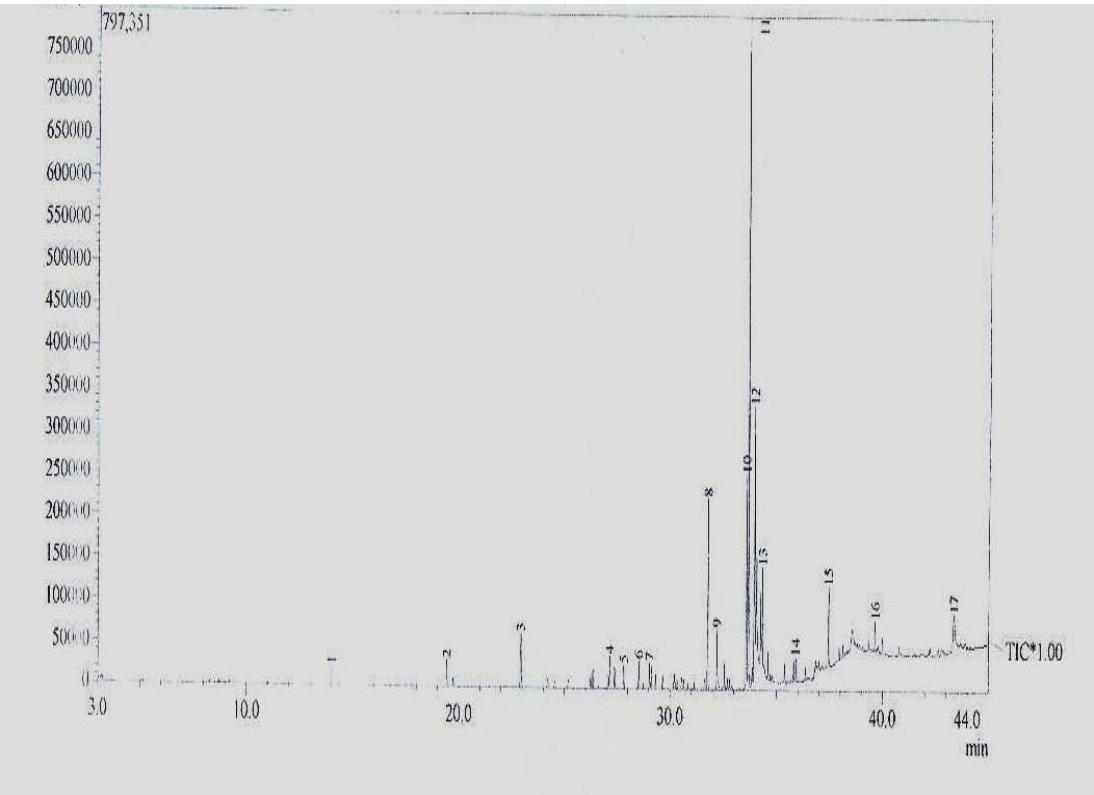

Figure 1. GC-MS spectrum of ethanol extract of *Andrographis paniculata*

The retention time, molecular formula, mass peaks, fragmentation peaks and concentrations of major fractions obtained from reliable spectral library are presented in Table 1. From the results obtained, it is evident that the separated compounds can be classified into hydrocarbons ((E)-tetradec-3-ene), alcohol (6-methyl heptan-1-ol), aromatic (undecan-5-ylbenzene, dodecan-5ylbenzene, undecan-2-ylbenzene, (2-methylundecan-6-yl)benzene and dioctyl phthalate).

(E)-tetradec-3-ene (1-Tetradecene) constitute about 2 % of the chemical constituent of *Andrographis paniculata*. It is an hydrocarbon that enters into all reactions typical of alpha olefins. 1-Tetradecene can be used in the production of amines and amine oxides, oxo alcohols, alkylated aromatics, alpha olefin sulfonates, epoxides, tanning oils and synthetic fatty acids. 6-methyl heptan-1-

ol or *n*-butyl alcohol is a primary alcohol constituting about 3% of the chemical constituent of *Andrographis paniculata*..

Table 1. Characterization of peaks obtained from GC-MS spectrum of ethanol extract of Andrographis *paniculata*

Line	Concentration (%)	Chemical formula	Retention time (s)	Mol. Weight (g/mol)	Name of compound	Mass peak	Fragmentation peaks
1	1.66	$C_8H_{18}O$	14.008	130	6-methylheptan-1-ol	8	55(95%), 69(100%), 97(50%)
2	2.21	$C_{14}H_{28}$	19.433	196	(E)-tetradec-3-ene	12	55(100%),69(70%), 83(60%), 97(50%), 111(25%)
3	4.42	$C_{14}H_{22}O$	22.933	206	3,5-di-tert-butylphenol	18	57(100%), 191(90%), 206(20%)
4	1.38	$C_{17}H_{28}$	27.150	232	undecan-5-ylbenzene	8	55(10%), 91(100%), 105(30%)
5	0.28	$C_{17}H_{28}$	27.817	232	dodecan-5-ylbenzene	7	55(10%), 91(100%), 119(40%)
6	1.10	$C_{17}H_{28}$	28.525	232	undecan-2-ylbenzene	8	55(10%),91(25%), 119(100%)
7	11.60	C18H30	29.033	246	(2-methylundecan-6-yl)benzene	7	55(10%), 91(100%),105(30%)
8	4.97	$C_{17}H_{34}O_2$	31.775	270	methyl palmitate	34	55(40%), 74(100%), 87(60%)
9	2.21	$C_{16}H_{32}O_2$	32.208	256	pentadecanoic acid	25	55(80%), 60(100%), 73(100%), 85(50%)
10	17.96	$C_{21}H_{38}O_2$	33.575	322	methyl icosanoate	45	55(65%), 67(100%), 81(65%), 95(55%), 109(35%)
11	18.78	$C_{19}H_{36}O_2$	33.667	296	methyl stearate	77	55(100%), 69(70%), 74(50%), 97(40%)
12	8.29	$C_{19}H_{38}O_2$	33.950	298	methyl stearate	36	55(30%), 74(100%), 87(70%)
13	6.63	$C_{20}H_{38}O_2$	34.325	310	ethyl stearate	33	55(100%), 69(70%), 83(50%), 88(55%),101(50%)
14	1.66	$C_{18}H_{35}NO$	35.892	281	oleamide	18	55(30%), 59(100%), 72(55%)
15	2.49	$C_{24}H_{38}O_4$	37.442	390	dioctyl phthalate	33	55(45%), 57(100%0, 71(70%),149(95%), 167(30%)
16	5.52	$C_{24}H_{48}O_2$	39.633	368	ethyl docosanoate	28	55(50%),71(30%), 88(100%0,101(90%0,396(30%)
17	8.84	$C_{15}H_{26}$	43.350	206	octahydro-1,4,9,9-tetramethyl-1H-3a,7-methanoazulene	38	53(80%), 65(70%), 79(65%), 91(100%), 105(80%), 119(90%)

The compound has a 4-carbon structure and the molecular formula. Its isomers include isobutanol, 2-butanol, and *tert*-butanol. Butanol is one of the group of "fusel alcohols" (from the German for "bad liquor"), which have more than two carbon atoms and have significant solubility in water. The compound is used in butter, cream, fruit, rum, whiskey, ice cream and ices, candy, baked goods and cordials. The largest use of *n*-butanol is as an industrial intermediate, particularly for the manufacture of butyl acetate.

2,6-Di-*tert*-butylphenol is the major fraction in line 3, constituting above 4% of the chemical constituents of the studied plant extract. The compound is colourless solid alkylated phenol and its derivatives are used industrially as UV stabilizer and as an antioxidant for hydrocarbon-based products ranging from petrochemicals to plastics. Illustrative of its usefulness, it prevents gumming in aviation fuels. 6-Di-*tert*-butylphenol is also a precursor to more complex compounds used as antioxidants and light-protection agents. In lines 4 to 7, closely related aromatic compounds were identified, The compounds included, (1-butylheptyl) benzene, (1-ethylnonyl) benzene, (1-methyldecyl) benzene and 91-pentylheptyl) benzene. The major similarity in these compounds is that they are aromatic compounds built from different hydrocarbon chains attached to benzene ring.

Lines 8 to 13 were characterized by several carboxylic acids which included, palmitic acid (lines 8 and 9), eicosadienoic acid (line 10) and octadecanoic acid (lines 11,12 and 13). The concentrations of these acids in the extract was found to ranged from 2 to 19 % and forms the dominant constituents of the plant extract. Industrial application of carboxylic acids cannot be overemphasized. Carboxylic acids are used in the production of polymers, pharmaceuticals, solvents, and food additives.

In line 14, the major constituent is 9-octadeceamide (2%), which is a Crodamide. Crodamide is the trade name of the range of fatty acid amides produced by Croda. Crodamides have a wide range of applications and are particularly used for solving issues with polymer processing and end use. In line 15, di-n-octyl phthalate is the major compound. This compound has various industrial utilizations. It is a commonly used plasticizer. It is also used as an additive to adhesives or printing inks. It is soluble in various organic solvents, e.g. in alcohol, ether and benzene. DBP is also used as an ectoparasiticide and in cosmetics and nail polishes.

In line 16, decasanoic acid was identified. The compound is used in intermediates for lithium docosanoate, silver docosanoate, other metal salt, docosylamine and higher alkyl esters. It is also used as plasticizers and stabilizers in the polymer industries. In line 17, 1H-3a,7-merhanoazulene is the major compound identified from GC-MS analysis. This compound is a building block and intermediate for pharmaceutical applications.

Figure 2 shows that the active compounds include, compounds 1, 8, 9, 10, 11, 12, 13, 14, 15 and 16.

3.2. Corrosion inhibition study

3.2.1. Effect of ethanol extract of Andrographis Paniculata

Figure 3 shows the variation of weight loss with time for the corrosion of mild steel in 0.1 M HCl in the absence (inserted) and presence of various concentrations of ethanol extract of *Andrographis Paniculata*. From the Figure, it is evident that weight loss of mild steel for the blank solution is higher than those obtained for solutions of HCl, containing ethanol extract of *Andrographis Paniculata*. This indicates that ethanol extract of *Andrographis Paniculata* inhibited the corrosion of mild steel in solutions of HCl. The figure also reveal that weight loss of mild steel increases with increase in the period of contact but decreases with increase in the concentration of the inhibitor indicating that ethanol extract of *Andrographis Paniculata* is an adsorption inhibitor. For an adsorption inhibitor, the inhibition efficiency of the inhibitor is expected to increase with increasing concentration, as observed in the presence study [13].

Table 2 shows the corrosion rates of mild steel and inhibition efficiency of ethanol extract of *Andrographis Paniculata* in various media. From the results, it can be seen that the corrosion rates of mild steel decrease with increase in the concentration of ethanol extract of *Andrographis Paniculata* but decreases with increasing temperature. Also, the inhibition efficiencies of various concentrations of ethanol extract of *Andrographis Paniculata* increased with increasing concentration but decreased with increase in temperature. Therefore, the adsorption of ethanol extract of *Andrographis paniculata* on

mild steel surface supports the mechanism of physical adsorption. For a physical adsorption mechanism is characterized by decrease in the value of inhibition efficiency with temperature [14].

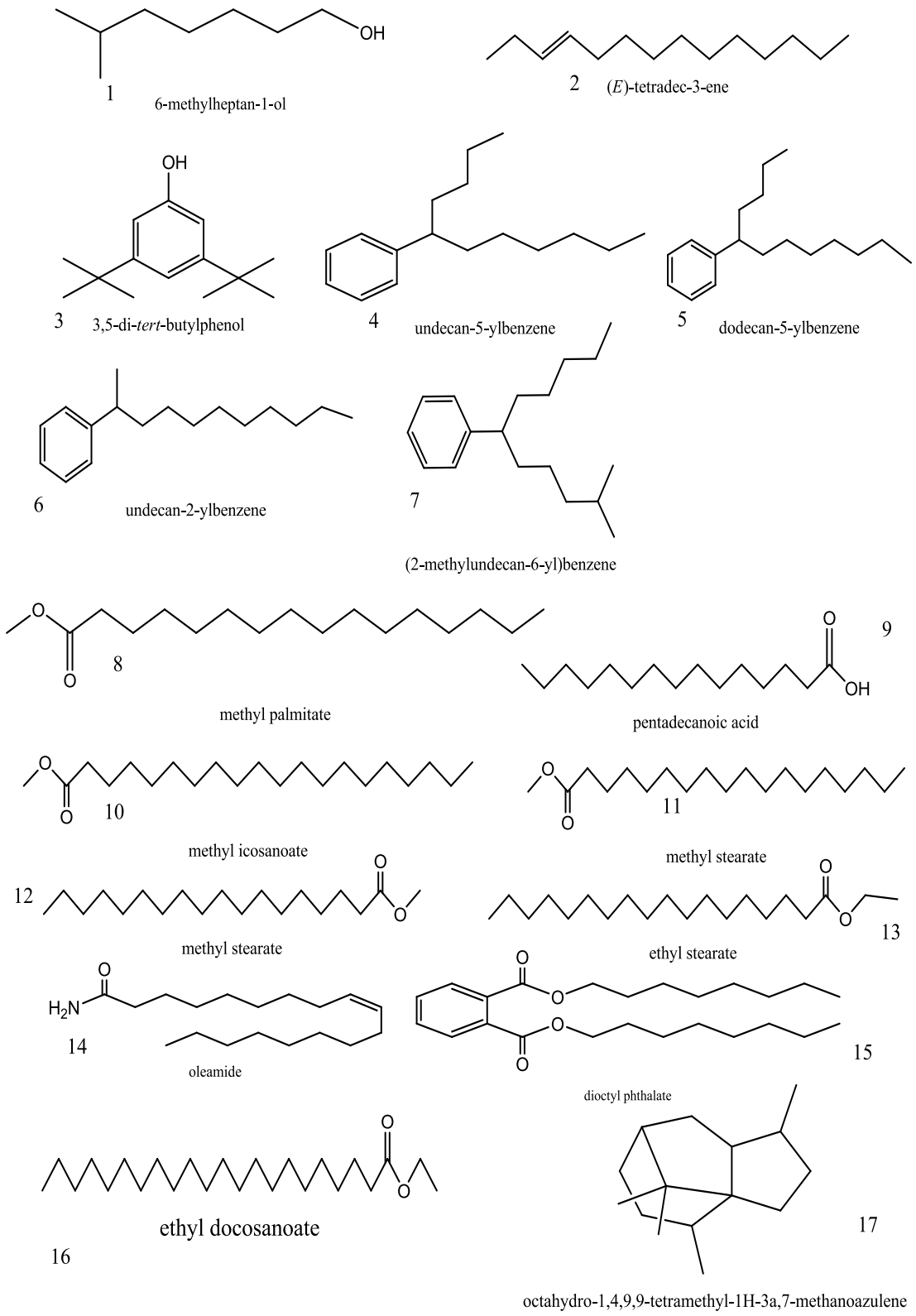

Figure 2. Chemical structures and labelling of molecules in ethanol extract of *Andrographis paniculata*

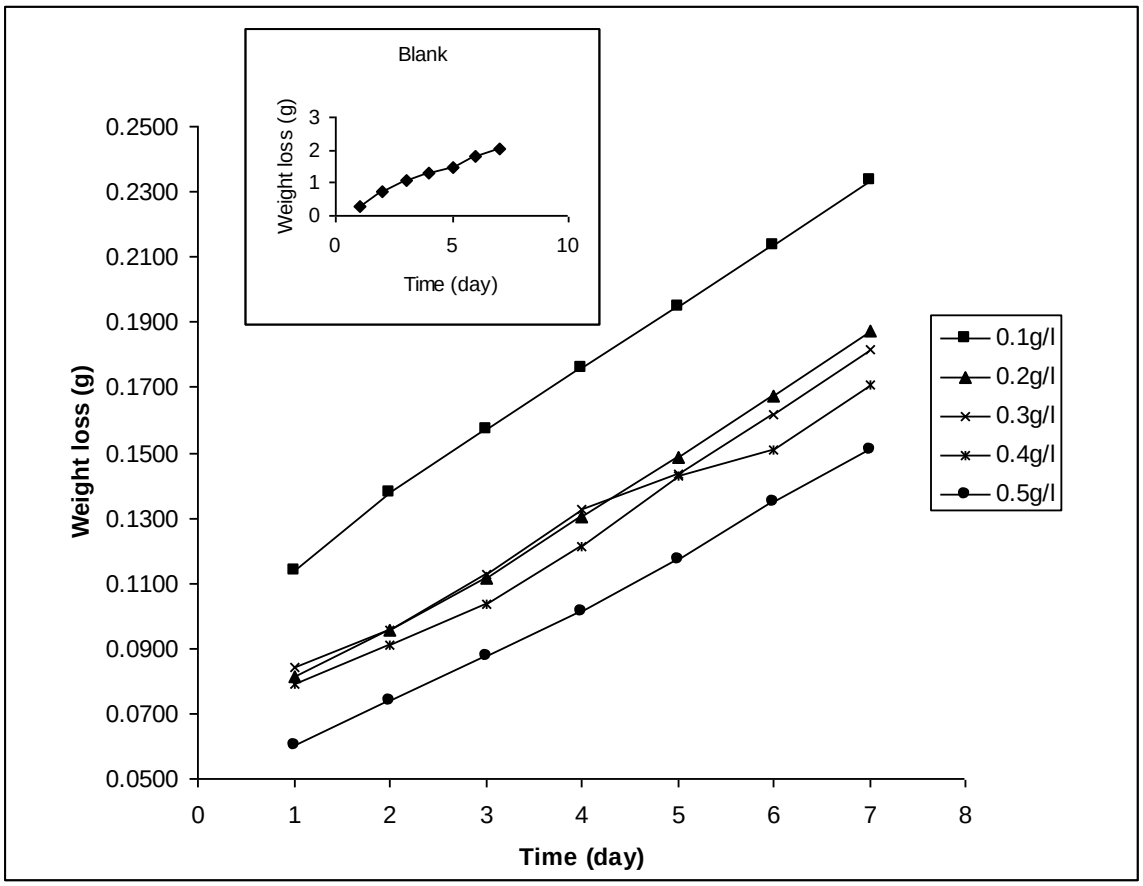

Figure 3. Variation of weight loss with time for the corrosion of mild steel in 0.1 M HCl in the absence (inserted) and presence of various concentrations of ethanol extract of *Andrographis Paniculata*

Table 2. Corrosion rates of mild steel and inhibition efficiencies of various concentrations of ethanol extract of *Andrographis Paniculata* in various media

C (g/l)	IE (303 K)	IE (333 K)	θ (303 K	θ (333 K)	CR x 10^{-4} (303 K)	CR x 10^{-3} (333 K)
0.0	-	-	-	-	6.08	43.50
0.1	88.57	68.34	0.8857	0.6834	0.70	40.53
0.2	90.85	72.44	0.9085	0.7244	0.56	11.99
0.3	91.12	78.33	0.9112	0.7833	0.54	9.43
0.4	91.64	82.01	0.9164	0.8201	0.51	7.83
0.5	92.63	85.22	0.9263	0.8522	0.45	6.43

3.3. Kinetic study

Kinetic study of the corrosion of mild steel in solutions of HCl containing various concentrations of *Andrographis paniculata* was studied by fitting data into various kinetic model and it was found that a plot of –log(weight loss) versus time gave a straight line indicating that the corrosion

of mild steel in the absence and presence of ethanol extract of *Andrographis paniculata* is consistent with the following equation [15],

$$-\log(\text{weight loss}) = -k_1 t/2.303 \quad (4)$$

where k_1 is the first order rate constant and t is the time.

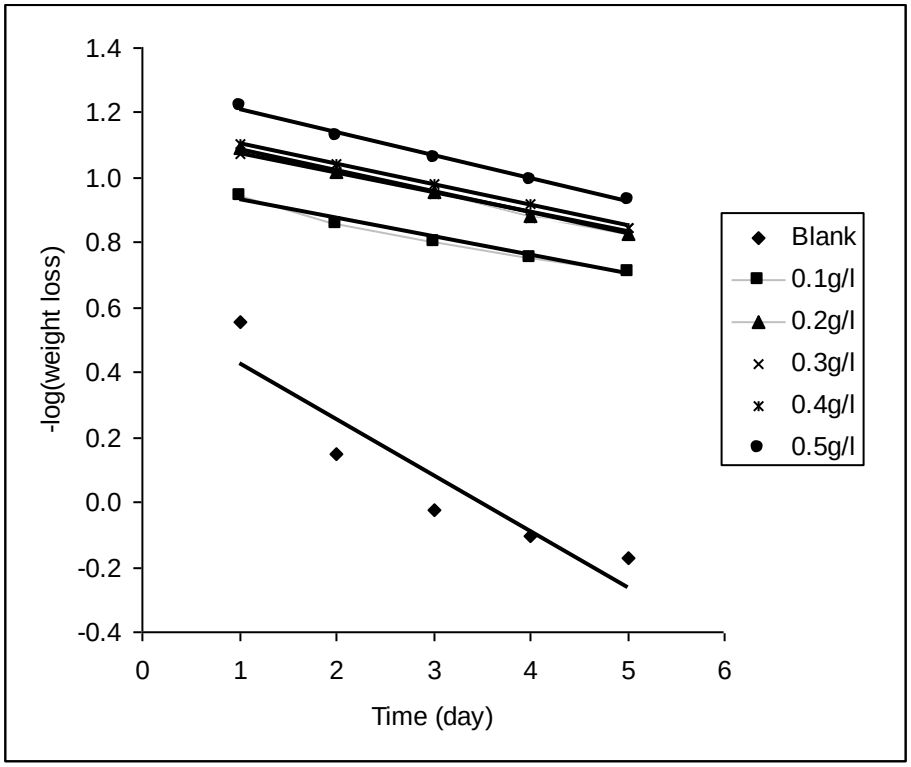

Figure 4. Variation of –log(weight loss) with time for the corrosion of mild steel in 0.1 M HCl containing various concentrations of ethanol extract of *Andrographis Paniculata*

Table 3. Kinetic parameters for the adsorption of ethanol extract of *Andrographis Paniculata* on mild steel surface.

C (g/l)	Slope	k_1	R^2	$t_{1/2}$ (day)
0.0	-0.1719	0.3959	0.8625	1.8
0.1	-0.0570	0.1313	0.9823	5.3
0.2	-0.0658	0.1515	0.9986	4.6
0.3	-0.0604	0.1391	0.9892	5.0
0.4	-0.0639	0.1472	0.9982	4.7
0.5	-0.0713	0.1642	0.9948	4.2

Figure 4 presents a plot of –log(weight loss) with time) for the corrosion of mild steel in solutions of HCl, containing various concentrations of ethanol extract of *Andrographis paniculata*.

Values of the rate constant (k_1) and degree of linearity (R^2) deduced from the plots are presented in Table 3. The closeness of R^2 values to unity indicates that equation 4 is applicable to inhibition of the corrosion of mild steel in solutions of HCl by *Andrographis paniculata*. Also, for a first order reaction, the half life is related to the rate constant as follows, [16],

$$t_{1/2} = 0.693/k_1 \quad (5)$$

calculated values of $t_{1/2}$ are also presented in Table 2. From the results obtained, $t_{1/2}$ for the blank solution is greater than those obtained for solutions of HCl containing various concentrations of *Andrographis paniculata* implying that ethanol extract of *Andrographis paniculata* increases the half life of mild steel in solutions of HCl.

3.4. Adsorption/thermodynamics considerations

The adsorption characteristic of the ethanol extract of *Andrographis paniculata* on mild steel surface was investigated by fitting data into different adsorption isotherms, including, Langmuir, Freundlich, Temkin, Frumkin, Flory-Huggins and El Awardy adsorption isotherms. The test revealed that the adsorption of ethanol extract of *Andrographis paniculata* on mild steel surface is best described by Langmuir adsorption model, which can be expressed as follows [17],

$$\theta = b_{ads}C \times 1/(1+K_{ads}C) \quad (6)$$

where θ is the degree of surface coverage of the inhibitor, b_{ads} is the equilibrium constant of adsorption and C is the concentration of the inhibitor in the bulk electrolyte. On rearranging equation 6, equation 7 was obtained and upon simplication, equation 8 was obtained,

$$C/\theta = C + 1/b_{ads} \quad (7)$$

$$\log(C/\theta) = \log C - \log b_{ads} \quad (8)$$

Figure 5 shows the Langmuir isotherm for the adsorption of ethanol extract of *Andrographis paniculata* on mild steel surface. Values of adsorption parameters deduced from the plots are recorded in Table 4. The results obtained indicate that the slope and intercepts are very close to unity. Therefore, the adsorption of ethanol extract of *Andrographis paniculata* on mild steel surface strongly adheres to the Langmuir adsorption model.

The equilibrium constant of adsorption obtained from the Langmuir adsorption isotherm is related to the standard free energy of adsorption according to the following equation,

$$\Delta G^0_{ads} = -2.303RT\log(55.5*b_{ads}) \quad (9)$$

where ΔG^0_{ads} is the standard free energy of adsorption of ethanol extract of *Andrographis paniculata* on mild steel surface, R is the universal gas constant and b_{ads} is the equilibrium constant of adsorption. The equilibrium constant of adsorption calculated from equation 9 are also presented in Table 4.

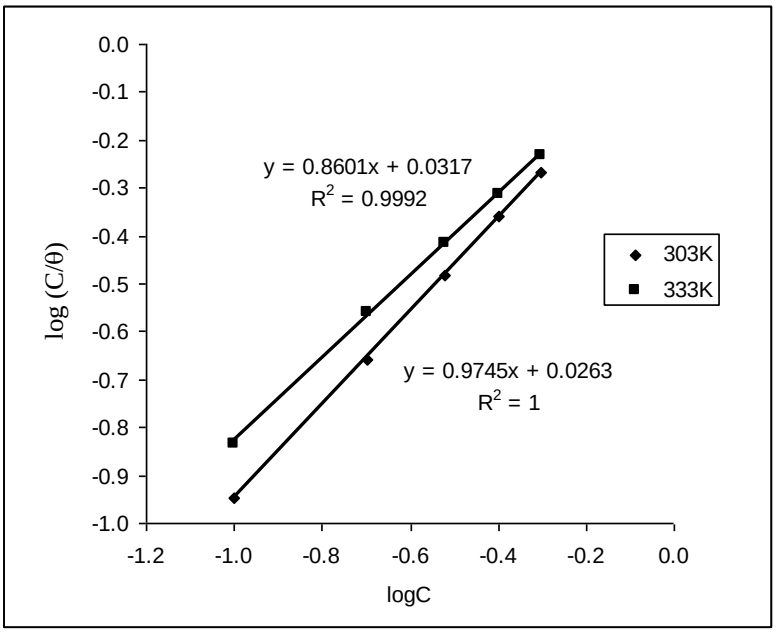

Figure 5. Langmuir isotherm for the adsorption of ethanol extract of *Andrographis paniculata* on mild steel surface at 303 and 333 K.

Table 4. Langmuir adsorption parameters for the adsorption of ethanol extracts of *Andrographis paniculata* leaves on mild steel surface

T (K)	Slope	log K_{ads}	R^2	ΔG^0 (kJ/ mol)
303	0.9745	0.0263	1.000	-10.25
333	0.8601	0.0317	0.992	-10.29

Calculated values of ΔG^0_{ads} are within the range of values expected for the mechanism of physical adsorption. Therefore, the adsorption of ethanol extract of *Andrographis paniculata* on mild steel surface is consistent with the mechanism of charge transfer from the inhibitor's molecule to the metal surface, which supports physical adsorption [18,19]

3.5. Mechanism of inhibition: Quantum chemical approach

From corrosion inhibition point of view, the compounds that are likely to be involved in the inhibition of the corrosion of mild steel by ethanol extract of *Andrographis paniculata* are those ones

that have hetero atoms in their long carbon or aromatic systems. From Figure 2, it is evident that the active compounds include compounds 1, 8, 9, 10, 11, 12, 13, 14, 15 and 16.

Table 5. Frontier molecular orbital energies of active constituents of ethanol extract of *Andrographis paniculata* responsible for corrosion inhibition

Molecule label	IUPAC name	E_{HOMO} (eV)	E_{LUMO} (eV)	ΔE (eV)
1	6-methylheptan-1-ol	-8.319	-0.405	7.914
8	methyl palmitate	-7.991	-1.490	6.501
9	pentadecanoic acid	-8.166	-1.740	6.426
10	methyl icosanoate	-7.910	-1.487	6.423
11	methyl stearate	-7.973	-1.488	6.485
12	methyl stearate	-7.973	-1.488	6.485
13	ethyl stearate	-7.937	-1.443	6.494
14	oleamide	-7.053	-1.589	5.464
15	dioctyl phthalate	-7.942	-3.384	4.558
16	ethyl docosanoate	-7.866	-1.446	6.420

At this juncture, we can invoke quantum chemical principles to investigate the order of importance of these compounds in the corrosion inhibition process. This is because, according to the frontier molecular theory, the reactivity of a molecular species can be correlated with the energies of the frontier molecular orbitals [20]. In Table 5, we present values of frontier molecular orbital energies of the ten compounds that are relevant in corrosion inhibition of mild steel by ethanol extract of *Andrographis paniculata*. The energy of the highest occupy molecular orbital (E_{HOMO}) is related to the tendency of a molecular species to donate electron while the energy of the lowest molecular orbital is associated with the the tendency of a molecular specie to accept electron [21]. Therefore, inhibition efficiency of a corrosion inhibitor is expected to increase with increase in the value of E_{HOMO} and a corresponding decrease in the value of E_{LUMO} [22]. On the other hand, the energy gap of a molecule (ΔE) is associated with the harness or softness of that molecule. A soft molecule is more reactive that a hard molecule and it is characterized with lower values of ΔE. Therefore, the , inhibition efficiency of a molecule is expected to increase with decreasing value of ΔE. In Fig. 6, the trend for the variation of the frontier molecular orbital parameters for the corrosion inhibition active compounds (in *Andrographis paniculata*) are presented. The presented trend is also expected to show the trend for the contribution of the various active components towards the inhibition of the corrosion of mild steel in solution of HCl. From the results obtained presented in Table 5 and Fig. 6, it is evident that the most active compound in the corrosion inhibition process is compound 5, which is pentadecanoic acid while the least active compound is compound 1, which is 6-methylheptan-1-ol . It is also evident from the results obtained that the values of the frontier molecular orbital energies for the ten active compounds are very close to each other indicating that the inhibition potential of *Andrographis paniculata* for mild steel corrosion is due to synergistic effect of the ten active compound.

4. CONCLUSIONS

From our study, we have found that ethanol extract of *Andrographis paniculata* is an adsorption inhibitor for the corrosion of mild steel in solution of HCl. The adsorption characteristics of the inhibitor favours the mechanism of charge transfer from the charged inhibitor's molecule to the charged metal surface (Physical adsorption) and supported the Langmuir adsorption model. Pentadecanoic acid, 6-methyl hepta-1-ol, methyl palmitate, methyl icosanoate, methyl stearate, oleamide, dicotyl phthalate and ethyl docosanoate are active ingredient in ethanol extract of *Andrographis paniculata* that synergistically interact to inhibit the corrosion of mild steel in solution of HCl.

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